# Optical analysis of ink and other contaminants in process waters

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**ABSTRACT:** Analysis methods developed specifically to determine the presence of ink and other optically active components in paper machine white waters or other process effluents are not available. It is generally more interesting to quantify the effect of circulation water contaminants on end products. This study compares optical techniques to quantify the dirt in process water by two methods for test media preparation and measurement: direct process water filtration on a membrane foil and low-grammage sheet formation. The results show that ink content values obtained from various analyses cannot be directly compared because of fundamental issues involving test media preparation and the varied methodologies used to formulate the results, which may be based on different sets of assumptions. The use of brightness, luminosity, and reflectance and the role of scattering measurements as a part of ink content analysis are discussed, along with fine materials retention and measurement media selection. The study concludes with practical tips for case-dependent measurement methodology selection.

***Application:*** Taking account of fines retention on measurement media and parameters such as scattering makes a significant difference on how reliable the results of process water quality analysis can be considered.

In mill processes, the accumulation of detrimental particles in process waters has been shown to have an impact on the quality of the end product, including pitch components, inks and other contaminants [1-8]. The long-term impact of small-scale contaminants can be severe because of the closure rate and solid buildup in water loops, both of which can have adverse effects on runnability and product quality [9-18]. The appearance of paper products relies on optical measurements, because they depict the way the end-user perceives the product quality. The assessment of optical parameters (brightness, whiteness, opacity, color, etc.) has been thoroughly reviewed by many literature sources [1,19-23].

The characteristics of pulp components can be measured from process waters using conventional methods. The content of solid and colloidal substances is commonly evaluated based on filtration or evaporation methods, wood extractives, and stickies with extraction methods, turbidity, organic carbon analysis, or even flow cytometry [24-30]. Analysis methods depicting the amount of ink and other components present that have specific adverse effects on the optical appearance of paper are not available. Such a method would be required to analyze the efficiency of different process water purification schemes on paper quality and paper machine cleanliness [2,15-18,31].

The amount of ink is often measured from pulp because of its near infrared (NIR) light absorption [32-35], either as the effective residual ink concentration (ERIC) at 950 nm or a similar measurement of residual ink (RI) at 700 nm. These methodologies have previously been adapted to measure ink content from mixtures of process water fines and fibers. For example, recent studies have used opaque sheets of 1.2 g (60 g/m$^2$) formed from different pulp and process water mixes on varying screens [31,36] using recirculation [2] or made on filter paper [2,3] to increase fine materials retention. Similarly, ink has been measured using an opaque 0.2 g (210 g/m$^2$) pad filtered on high-retention membrane foil [37]. Measurement of the scattering coefficient, low-grammage sheets of approximately 30 g/m$^2$ was achieved on 1-2 µm filter paper from virgin fibers and process waters [17,37,38]. Despite the variety of methods currently available, no single, well-defined protocol exists. There are open questions related to measurement of media preparation and the utilization of measured scattering coefficient.

This study is designed to clarify the complexities involved when assessing the effect white water contaminants have on the optical appearance of paper. Different optical measurement methodologies are used and compared for their benefits and limitations in measuring process water fine materials. Additionally, the role of parameters such as ISO brightness (457 nm), luminosity (557 nm), or intrinsic reflectance value (700 nm) are discussed. As a practical conclusion, suitable methods for different white water analysis requirements are proposed based on their operational characteristics.

## MATERIALS AND METHODS

### *Determination of optical properties*

Preparation of pads on a membrane

Direct filtration of process water was used to prepare pads on a membrane. The method was comparable to normal pad preparation [37], except the pads were prepared by filtrating process water equivalent to 0.2 g of dry mass on a membrane foil (Sartorius ø 50 mm, pore size 0.45 µm; Sartorius AG, Goettingen, Germany), resulting in a relative pad grammage

of approximately 210 g/m². The pH was not adjusted. The prepared pads were considered optically opaque based on earlier studies [37,39]. The drainage rate for samples differed in the range of 10-15 min, depending on the process water solids content. Pads on membrane foils were dried in a desiccators. Optical properties were measured from pulp pads using an L&W Elrepho spectrophotometer (Lorentzen & Wettre; Kista, Sweden). The results were compared to membranes on which 100% deinked pulp (DIP) was filtered. The reference pulp optical properties were: luminosity, 64.4%; reflectance at 700 nm, 68.4%; ISO brightness, 60.1%; and ink content, 433 ppm.

Preparation of low-grammage sheets on filter paper

Sheets with a grammage of 30 g/m² were prepared by mixing a constant amount of fibers (hyperwashed virgin supercalendered grade denoted as HWSC) with process water to ensure strength and slight transparency (opacity < 97%) of the prepared sheet. The pH was not adjusted. The fibers used for mixing were obtained by hyperwashing unprinted SC grade material. The mixing ratio of 70% fibers and 30% process water fines was kept constant for all the experiments and calculated based on dry masses of 0.82 g for sheets of 165 mm by 165 mm. Hence, 0.57 g of fibers and a volume of process water containing 0.25 g of solids were mixed and diluted with deionized water to approximately 250 ml. Sheets were prepared in a sheet mold on filter paper (Munktell 00H; Munktell Filter AB, Falun, Sweden) having a porosity of 1-2 µm for high retention. Drainage rate for the prepared 250 ml samples differed within the range of 5 to 10 min. A more detailed description of the method is given in [37]. The results were compared to low grammage sheets of 100% hyperwashed SC pulp. The optical properties of HWSC reference pulp were: luminosity, 75.4%; reflectance at 700 nm, 87.0%; ISO-brightness, 70.4%; and ink content measured as 36 ppm, although the virgin pulp contained no ink.

Optical measurements

Optical brightness and luminosity were tested according to SFS-ISO 2470 "Paper, board and pulps. Measurement of diffuse blue reflectance factor (ISO brightness)" and TAPPI T 527 om-02 "Color of paper and paperboard (d/0, C/2)." A test media absorption coefficient (k) was determined using the scattering coefficient (s) and reflectance ($R_\infty$) of an opaque stack, according to Eq. (1). The scattering coefficient was measured from sheets according to Eq. (2). A constant scattering value of 42 m²/kg, given in INGEDE Method 2 "Measurement of optical characteristics of pulps and filtrates from deinking processes," was also used. Reference measurements were taken from the pads and sheets without addition of white water. All measurements were performed at 700 nm.

$$k = \frac{s \cdot (1 - R_\infty)^2}{2 \cdot R_\infty} \quad (1)$$

$$s = \frac{R_\infty}{w \cdot (1 - R_\infty^2)} \cdot \ln \frac{R_\infty \cdot (1 - R_0 \cdot R_\infty)}{(R_\infty - R_0)} \quad (2)$$

where k is the absorption coefficient at 700 nm [m²/kg],
s the scattering coefficient at 700 nm [m²/kg],
w is the grammage of the sheet [kg/m²],
$R_0$ is the reflectance against a black cavity at 700 nm,
$R_\infty$ is the reflectance of the stack at 700 nm.

Analyzed white waters

The process waters studied comprised three process waters collected from mill sites and prepared model water samples. Water sample 1 had components from both thermomechanical pulp (TMP) and DIP in a feed ratio of 35%/65%, denoted here as WW1. Water samples 2 and 3 came from mills that run 100% deinking pulp-based processes. This series is denoted as WW2 and WW3. The samples were treated with flotation to obtain varied levels of ink and fine material. The pH, suspended solids content, and the amount of inorganic ash from all solid materials present in the process waters were analyzed (SFS-EN 872 "Determination of suspended solids," 2005; and ISO 1762 "Paper, board and pulps – determination of residue [ash] on ignition at 525°C," 2001). The results are presented in **Table I**.

Analyzed model waters

Prepared model waters involved the dissolution of varied fine materials into a water suspension solely to monitor the effect of changes in scattering properties on the ink content determination. The fines selected were typically used grades of ground calcium carbonate (GCC) and kaolin, obtained as commercial products, and TMP fines that were collected by

| ID | pH | Suspended Solids [%] | Ash 525°C [%] |
|---|---|---|---|
| WW1 | 7.5 | 0.2 – 2.2 | 52.7 – 70.2 |
| WW2 | 8.0 | 0.5 – 1.2 | 55.7 – 69.4 |
| WW3 | 8.2 | 2.0 – 3.6 | 73.5 – 76.3 |

***I. The pH and solids content of the mill process waters studied.***

| Solution No. | GCC [%] | Kaolin [%] | TMP Fines [%] |
|---|---|---|---|
| 1 | - | - | 100 |
| 2 | - | 100 | - |
| 3 | 100 | - | - |
| 4 | 33 | 33 | 33 |
| 5 | 25 | 25 | 50 |

***II. Mixing ratio of fine material in prepared process water solutions.***

| Fiber Material # | Printed | Attached Ink Content in HW ONP [ppm][1] |
|---|---|---|
| Fiber 1 | No | 73 |
| Fiber 2 | Yes | 202 |
| Fiber 3 | Yes | 422 |
| Fiber 4 | Yes | 632 |
| [1] Pulp ink contents measured from low-grammage sheets. | | |

***III. The components of test pulps used as fiber fractions in the scattering coefficient measurement studies.***

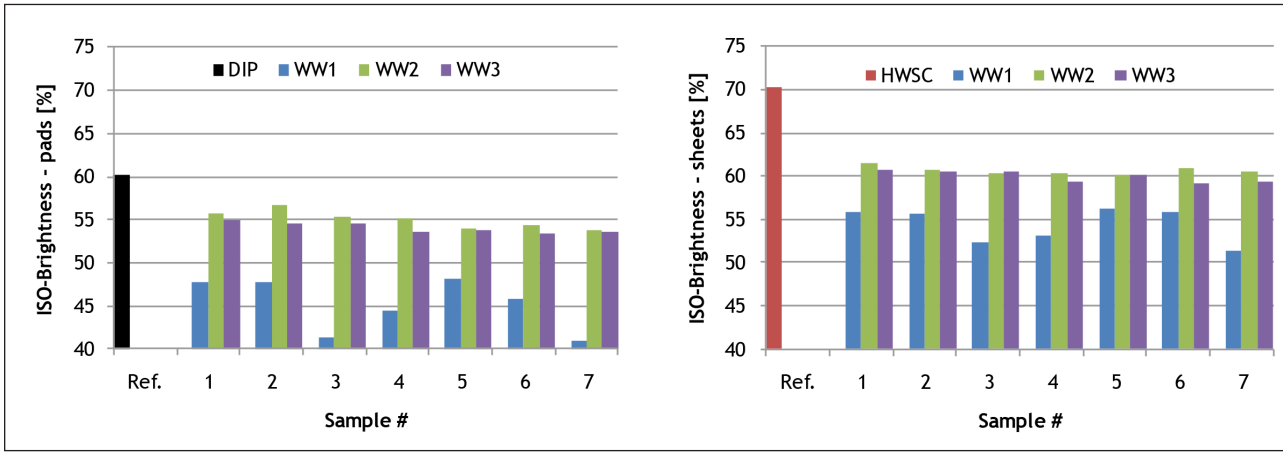
rinsing and thickening them from pulp through a 150 mesh wire screen. Mean particle sizes of fillers were 7.9 µm and 5.0 µm for kaolin and GCC respectively, as determined with a laser diffractometer (Beckman Coulter LS 13320; Beckman Coulter, Fullerton, CA, USA). From these, five different mixtures were prepared, as shown in **Table II**, where the proportions are shown on a dry mass basis.

The prepared model waters contained only fillers and fines. Their ink content was varied by using fiber material that contained fixed levels of ink. The sheet preparation was similar to the earlier process [37], but pulp from printed newspaper was used in addition to the same unprinted paper. Four pulps were prepared by oven drying at 60°C and hyperwashing the pulped ONP (cold-set web-offset printed old newsprint, HW ONP) samples with varying pulping times to obtain distinctly differing levels of attached ink content that were then mixed with different mixtures of fillers, as shown in Table II. The prepared pulps are summarized in **Table III**.

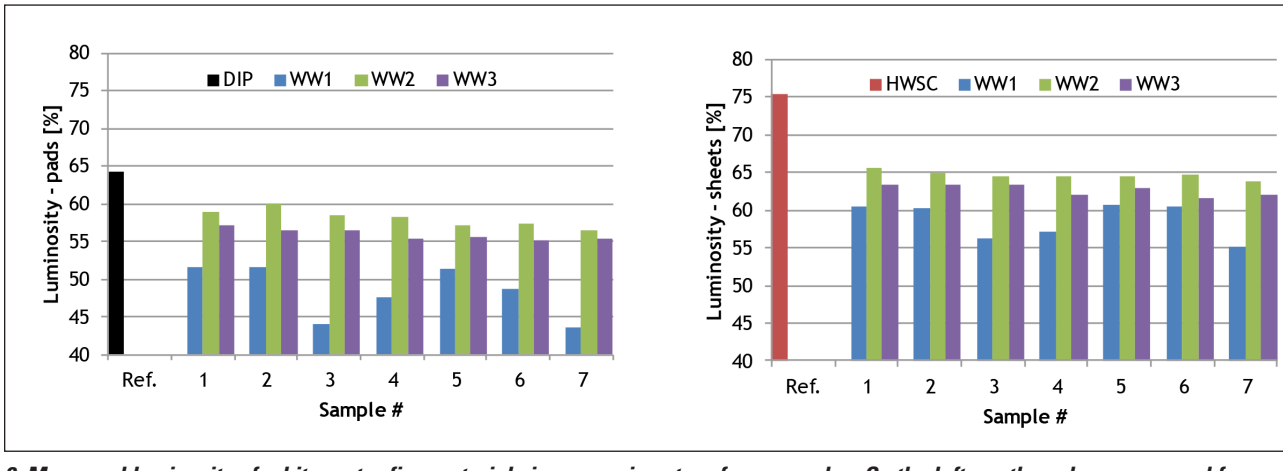
## RESULTS

### *Effect of water dirt content on the optical appearance of pulps*

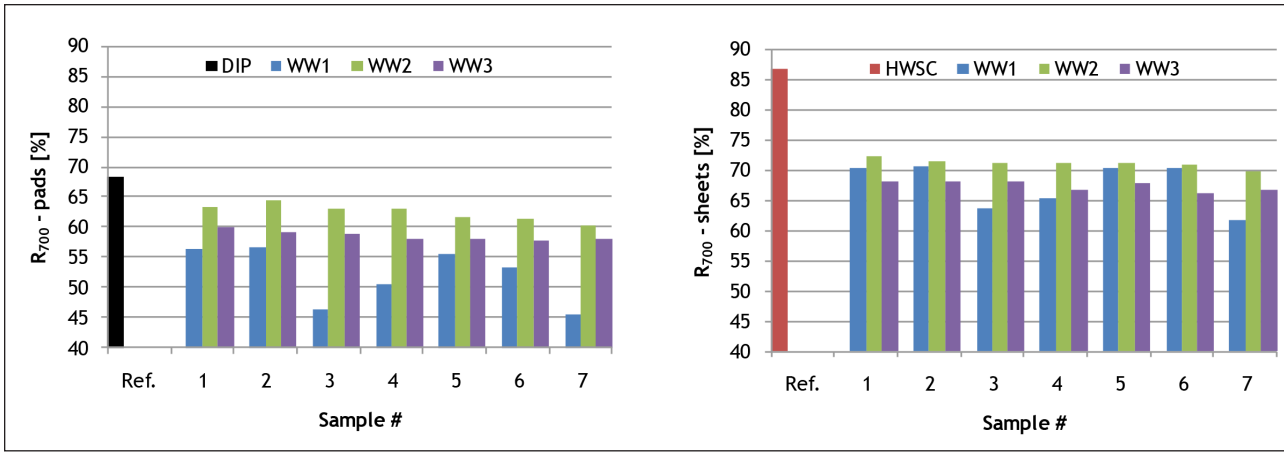
The impact of circulation water contaminants was compared directly from pads with the optical properties of a typical DIP and indirectly as their potential to darken or stain pure HWSC pulp in the case of low-grammage sheets. **Figures 1-4** illustrate the measured ISO-brightness, luminosity, reflectance at 700 nm, and ink content of white waters (WW1-WW3), which



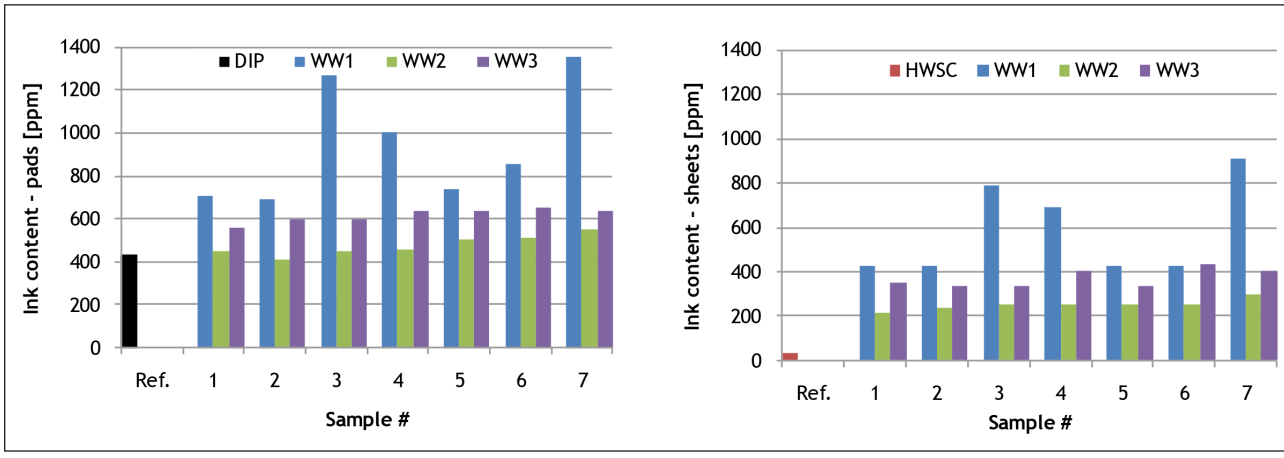

***1. Measured ISO brightness of white water fine materials in comparison to reference pulps. On the left are the values measured from opaque pads in comparison to 100% deinked pulp (DIP), and on the right, the darkening of low-grammage sheets (30% white water fine components) with respect to the hyperwashed virgin supercalendered (HWSC) grade is shown.***




***2. Measured luminosity of white water fine materials in comparison to reference pulps. On the left are the values measured from opaque pads in comparison to 100% DIP. On the right, darkening of low-grammage sheets (30% white water fine components) with respect to HWSC is shown.***




***3. Measured reflectance of white water fine materials in comparison to reference pulps. On the left are the values measured from opaque pads in comparison to 100% DIP. On the right, darkening of low-grammage sheets (30% white water fine components) with respect to HWSC is shown.***




***4. Measured ink content of white water fine materials in comparison to reference pulps at 700 nm using a constant scattering of 42 m²/kg. On the left are values measured from opaque pads in comparison to 100% DIP. On the right, darkening of low-grammage sheets (30% white water fine components) with respect to HWSC is shown.***

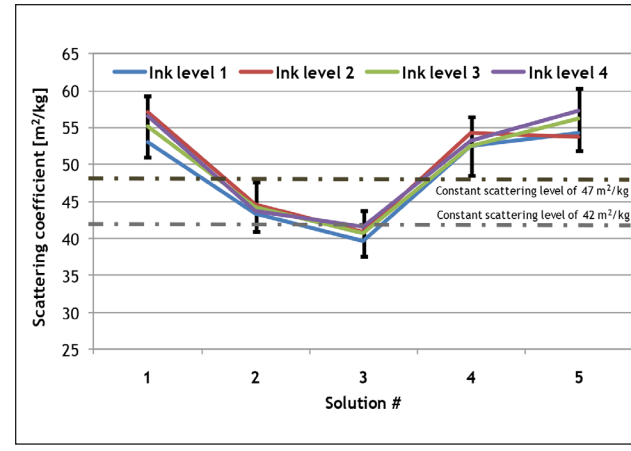
are also given in Table I. Both methods are shown to provide applicable results in the evaluation of the optical properties of white water fine materials.

As seen from Fig. 1, the white water solid components can be characterized differently, depending on the measurement media. A direct comparison of values is possible only from the pads on the left, showing that the brightness of the process water solid components is lower than that of the pulp pads. From the pads, the WW's 2 and 3 appear to have 5 to 8 percentage points lower brightness than DIP. WW1 has 14 to 18 percentage points lower ISO brightness. On the right, the mixes of waters and HWSC pulp have notably lower brightness values than the hyperwashed pulp itself. This observed decrease in brightness of the sheets remains a more indicative result because of the presence of fibers in addition to white water solids; however, differences in white water composition can be noted from either measurement method. The comparison given in Fig. 2 on measured luminosity shows an analogous effect to the aforementioned brightness values – mixing the "dirty" water fractions in HWSC pulp lowers the optical properties of the mixes. The values obtained from pads are also lower for white water components than for DIP. The amount of dirt in the water samples in comparison to pulps gives a similar result to brightness. The difference is most pronounced in the case of WW1, which has 13 to 20 percentage points lower luminosity than DIP. Corresponding figures for WW2 and WW3 are 4 to 7 and 7 to 9 percentage points, respectively. In the case of sheets, the fine materials in WW1 lower the observed luminosity by 15 to 21 percentage points; those in WW2, by 10 to 12 percentage points; and those in WW3, by 13 to 15 percentage points.

Reflectance at 700 nm shows a trend similar to the previously mentioned parameters, although it is more sensitive to inks. Measurement from opaque pads in Fig. 3 (left) shows that the white water fine components reduce the measured reflectance by 12 to 27 percentage points in the case of WW1, 4 to 8 percentage points in the case of WW2, and 8 to 10 percentage points in the case of WW3. When compared with

sheets, the difference appears to be 17 to 25 percentage points in the case of WW1, 14 to 17 percentage points for WW2, and approximately 20 percentage points for WW3.

The selectivity of ink content analysis (Fig. 4) makes the white water samples differ most significantly. White waters clearly appear to contain substantially more ink than DIP (433 ppm), changing the levels from 700 to 1370 in WW1, from 450 to 630 ppm in WW2, and from 580 to 630 ppm in WW3. On the right, HWSC has practically no ink; however, sheets containing 30% white water solids clearly show a level of pulp contamination rising up to 920 ppm for WW1. The ink content analysis is most selective in showing the differences between dark white water solid components other than fiber fines. However, the results given in Fig. 4 do not take into account the changes in their scattering properties. Differences in the ink content analysis are studied in the following section.



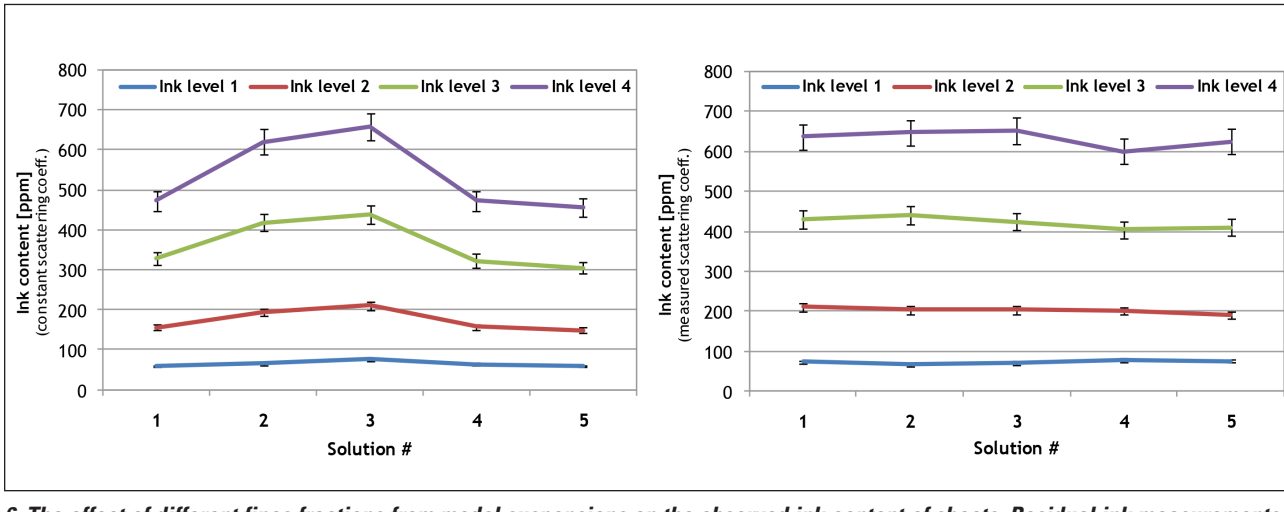

*5. The change in sheet scattering coefficient values measured at 700 nm with different fines fractions (30%) from model suspensions and different pulps as fiber fractions (70%) of the sheet. Error bars of 7.6% were used, as given in [38].*

### *Effect of the scattering coefficient on the measured ink content*

Substantial changes were noted in the NIR scattering of sheets, depending on the content of different components. Here, the ink content of samples 1-4 was kept constant by the use of model waters, including fiber components from the same base paper as unprinted (Fiber 1) and cold set printed ONPs (Fibers 2-4) with recorded levels of attached inks, while the scattering properties of pulp-fines mixes were changed (Tables II-III).

The scattering coefficients of the furnishes studied, comprising the unprinted fiber fraction, the added fiber fines (TMP), and the mineral fillers (kaolin clay and ground calcium carbonate [GCC]), are shown individually and in the mass weighed proportions used in **Fig. 5**. The alternative constant scattering level is also given as a reference. Results are presented with error bars at 7.6% based on the TAPPI repeatability ratio given in [38]. As seen, constant levels of scattering would in these cases give a poor correlation to the optical scattering properties of the suspensions when the fine materials' content of the white water changes, which would also lead to incorrect ink content estimation.

The ink content obtained from model waters using a constant scattering coefficient value of 42 m²/kg is shown in **Fig. 6** (left), with error bars at 5.1% based on [37]. The insensitivity to changes in fines scattering properties (Table II) is shown to have a considerable effect on the perceived ink content. Obtained values should be constant. The use of constant scattering implies that the ink content changes, although this is not the case. Taking the measured scattering into account allows a more accurate analysis of the ink content of the water sample, shown in Fig. 6 (right), where the ink levels appear to stay constant for each solution within the given error limits.




*6. The effect of different fines fractions from model suspensions on the observed ink content of sheets. Residual ink measurements are made with 700 nm from sheets using the constant scattering coefficient of 42 m²/kg (left) and the measured scattering coefficients (right). Error bars of 5.1% were used, as given in [37].*

## DISCUSSION

### Optical characterization of white water fine materials

Optical properties measured from membrane pads (left sides of Figs. 1-3) show the optical properties of the white water fine components when compared with 100% DIP. Direct comparisons can be made between the obtained brightness, luminosity, and reflectance at 700 nm. Similarly, the difference in white water solids differs when mixed with clean fiber and measured from sheets. However, in this case, a comparison between the obtained values cannot be made directly. The sheet method allows the comparison of different white waters to determine their relative purity, but it does not give absolute values for the properties of white water fine materials.

The importance of measured NIR scattering is not apparent from Fig. 4, where constant scattering is used, but the model waters analyzed in Fig. 5 show a remarkable difference. White water fines components can vary significantly in nature (Fig. 4). Figure 5 shows that constant scattering values are unable to take into account the changes in white water fine materials' content and scattering properties (15 units). As seen from Eq. (1), scattering affects light absorption and ink content significantly. For example, the use of a constant scattering coefficient of 42 kg/m$^2$ when the true value would be 55 kg/m$^2$ would yield a 30% bias in the measurement. This effect has previously been shown to apply to deinked pulps [37,38,40]. In practice, if white waters contain fines with high scattering properties, they can also hold substantial amounts of ink without necessarily appearing "dirty." In contrast, if the fines present in process waters have poor scattering properties, even small amounts of ink can lower the observed brightness and luminosity. The same reasoning also applies to reflectance, even if it is measured with wavelengths most sensitive to inks.

Figure 6 illustrates this effect and shows how the mixes of white waters and pulps are observed differently when determining ink content with either constant or measured scattering coefficients. The pulp mixes had constant ink contents, but the use of constant scattering was unable to compensate for the changes occurring in the composition of white water fine materials. The use of measured scattering values took these changes into account and provided a statistically accurate measure of the white water ink content, as previously shown for deinked pulps [38,40].

### Methods and test media

The comparison of methods and test media is complicated by the number of available methodologies for pulping analysis. Most commonly, measurements have been made from sheets or opaque pads, but the specifics of their preparation are only

| STRENGTHS | WEAKNESSESS |
|---|---|
| A: Easy and fairly simple method, results easily comparable. | A: Poor retention of fines because of the 25 µm filter paper aperture. Indirect measurement because of the presence of pulp. |
| B: Results easily comparable, good retention possible because of recirculation. | B: Very time-consuming (multiple sheets needed) and requires some monitoring of fine material retention. Indirect measurement because of the presence of pulp. |
| C: Easy and fairly simple method, results easily comparable. | C: Poor retention of fines because of the 11 µm filter paper aperture. Indirect measurement because of the presence of pulp. |
| D: Offers good retention of fines because of the 1-2 µm filter paper aperture and can be used to determine scattering coefficient changes for each sheet (opacity < 97%). | D: Indirect measurement because of the presence of pulp. |
| E: Quick and easy measurement from sample to result. High retention because of 0.45 µm membrane aperture. Can be used to compare optical properties directly. | E: None. |
| **OPPORTUNITIES** | **THREATS** |
| A: Sheets of 60 g/m$^2$ also usable for study of mechanical sheet properties. | A: Does not account for possible changes in scattering properties of white water fine materials. |
| B: Sheets of 60 g/m$^2$ also usable for study of mechanical sheet properties. | B: Does not account for possible changes in scattering properties of white water fine materials. |
| C: Sheets of 60 g/m$^2$ also usable for study of mechanical sheet properties. | C: Does not account for possible changes in scattering properties of white water fine materials. |
| D: Best for comparative studies between mills and for other research purposes. | D: If fiber source is hyperwashed, there is less fiber fines in the measurement sheet than in papermaking, thus highlighting the optical effect of white water solids. |
| E: Sufficient for day-to-day monitoring purposes on a production line. | E: Does not account for possible changes in scattering properties of white water fine materials. |

***V. SWOT analysis of recently applied methodologies for optical characterization of white waters.***

explained in broad terms. Their grammage is often not clearly discussed. Additionally, the retention, media characterization, and use of constant scattering can lack in accuracy. The following SWOT (strengths, weaknesses, opportunities, and threats) analysis (**Table V**) depicts some recent methodologies used for the optical analysis of white water properties. The abbreviations used are: **A**: 1.2 g (60 g/m²) sheet made on a 500-mesh wire [31]; **B**: recirculated 1.2 g (60 g/m²) sheet [2]; **C**: 1.2 g (60 g/m²) sheet on 11 µm filter paper [2,3], based on a similar method using a 20 µm wire screen [36]; **D**: low-grammage (30 g/m²) sheet made on 1-2 µm filter paper [17,37,41]; and **E**: 0.2 g (210 g/m²) pad filtered on a 0.45 µm membrane foil [37].

### *Remarks on test media and analysis*

Only a few qualities of the measurement media were important in the analysis: the retention of fine materials and the opaque nature of the test media. If the small particles that account for most of the ink and colloidal pitch are not retained in the media, the representativeness of the measurement remains poor [42]. The use of a small pore-size filter can be considered beneficial since this does not unreasonably prolong the drainage time. The use of white water recycling to obtain high retention can be problematic because it has been observed that, in practice, the true equilibrium is rarely reached in both ash and ink. Multiple sheets should be prepared and ashed to verify the equilibrium, which further adds to both the time and trouble taken for analysis. Opaque pads are proven to have high retention of small ink particles and can be considered beneficial, whether made on paper or membranes [39,41].

Similarly, if the changes in fines scattering cannot be determined, the analysis can become insensitive to ink content. Estimation of scattering is possible only from translucent sheets, but this often limits the amount of white water fine material that can be deposited onto the sheet without rupturing its structure prior to the measurement. In practice, some fiber components are needed to enforce the sheet matrix where they will contribute to the optical measurement result [37]. As the ratio of white water solids and pulp changes, the scattering also changes, but in a nonlinear manner [43]. Hence, the correct assessment of the absolute scattering of white water solid components is not possible using this method. However, low-grammage sheets can detect changes in scattering that can be related to changes in fine materials to obtain a measure of ink content change within process waters.

As a practical conclusion, it appears that the best way to monitor the changes in the properties of white water fine material is to produce opaque pads on small, pore-size membranes and compare either ISO brightness, luminosity, or reflectance values between white water samples. When ink content is of specific interest, the NIR scattering coefficient of the white water fine materials should be obtained from low-grammage sheets.

## CONCLUSIONS

The easiest and fastest way to analyze process water optical appearance is by making opaque pads on membrane foil to ensure high retention of small ink particles. This result represents the sum effect of all the components present in suspension and can be compared to pulp luminosity, ISO brightness, or reflectance. If the ink content is of specific interest, methods that can provide a quantitative measure of the NIR scattering coefficient are needed. This is achieved by production of sufficiently low-grammage sheets that meet the opacity requirement from mixes of process water and selected reference pulp. Hyperwashed, unprinted fibers provide one suitable matrix for these sheets, although DIP can also be applied. The results obtained can then be compared to pulp sheets to measure the difference caused by ink particles to the ERIC measured between pulp sheets and white water sheets. **TJ**

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We chose to research this topic because proposed methods to measure the optical quality of white water fines remained unclear in literature. We focused on the measurement of scattering coefficient and its importance, which has been shown to have an influence on the determination of ink content from pulps. This study complements earlier studies by introducing different sample preparation methods and assessing their differences and usability regarding the influence of white water quality on pulp and end-product properties.

The most difficult aspect of the study was to prepare pads and sheets consistently, because applicable TAPPI and INGEDE methods are not available for analyzing fines properties from water samples. It was interesting to observe how much ink and other fine materials were rinsed out from prepared sheets if prepared on, for example, wire screen without filter paper. Moreover, the lack of previous studies in this area made the work genuinely interesting and novel by nature.

Paper and board manufacturers using deinked material would benefit from this study by understanding how significantly the circulation waters may affect their processes, and also that selection of measurement method certainly impacts reliability and results.

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